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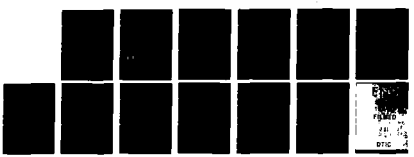

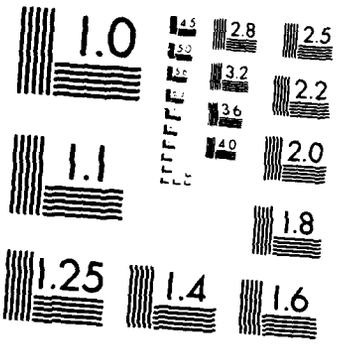

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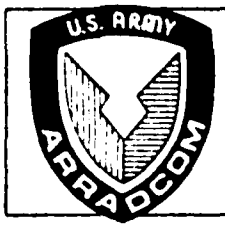
ANTHONY F. TASCHLER

JUNE 1983



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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

A round robin was conducted to statistically evaluate four chemical methods for determining the percentage of nitrogen in nitrocellulose. The four methods are: (1) nitrometer, (2) titanous chloride, (3) automatic ferrous sulfate titration, and (4) potentiometer ferrous sulfate titration. The nitrometer method was used as the standard and the other three were tested against it for equivalency. Automatic titration proved to be equivalent, while potentiometer titration has equivalency potential given more experience in and further

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20. ABSTRACT (cont)

development of the technique. The titanous chloride method is not equivalent to the nitrometer because the variance is too high.

ACKNOWLEDGMENT

The author wishes to acknowledge the contributions made by the personnel of the participating activities and the members of the Propellant Characterization Subcommittee of the Joint Army Navy NASA Air Force (JANNAF) Interagency Propulsion Committee. The author is also grateful to Eric R. Bixon of ARRADCOM for the statistical analysis of the data listed in this report.

CONTENTS

INTRODUCTION

A round robin was conducted to determine if any one of three proposed chemical methods for determining the nitrogen content of nitrocellulose was equivalent to the existing nitrometer method. The three proposed methods are: (1) potentiometer ferrous sulfate titration, (2) automatic ferrous sulfate titration, and (3) titanous chloride.

DISCUSSION

In the round robin, ten analyses were made of two samples[1] of nitrocellulose using four different methods for determining the percentage of nitrogen, specifically:

1. Nitrometer, Method 209.3
2. Titanous chloride, Method 209.7
3. Potentiometer ferrous sulfate titration, Method 209.11
4. Automatic ferrous sulfate titration

Methods 1 through 3 are from Mil Std 286; method 4, from Hercules, Inc. (refs 2, and 3).

The nitrometer method was chosen as the standard since it is currently used for acceptance testing of nitrocellulose in accordance with Specification Mil-N-244 and Mil Std 286.

Data Gathering

Tables 1 and 2 list the data gathered by different participating activities and the methods used in the round robin. The participating activities and the symbols (A, B, C, etc.) by which their laboratories are identified in the tables are listed below:

Participating activities	Symbol
Olin Corporation	A
Hercules, Incorporated	B

[1] The first sample (lot 15340) and the second sample (lot 30057) consisted of 5 pounds of grade A and grade C nitrocellulose, respectively, as specified in Mil-N-244 (ref 1).

Radford Army Ammunition Plant (RAAP)	C
ARRADCOM	D
Naval Ordnance Station	E
Edwards Air Force Base	F
Lawrence Livermore National Laboratory	G

The tables also list the averages, variance, and standard deviation of each laboratory and method.[2] The variance and standard deviation of the automatic method are equivalent to the nitrometer method. The data from the potentiometer method appear to depend on each individual activity; more experience with this method is needed.

The raw data from tables 1 and 2 are plotted in figures 1 and 2. A graphical comparison of the data is made by plotting all of the individual data points (represented by X's) and the mean of each set of ten data points (represented by lines). The graph is an ideal method for visualizing differences in the data. The nitrometer data appear to have about the same dispersion as the data from the automatic and potentiometer methods. The data from RAAP (C) show dispersion and test results for the three methods. The data from the titanous chloride method appear to be more dispersed than the other three.

Statistical Evaluation

The data were evaluated by a two-way analysis of variance, and the results are shown in tables 3 and 4. The values determined by the F-test (ref 4) at the 95% level are significant for the activities and interactions for the test methods. Repeatability and reproducibility tests were determined for each method. Since the values calculated for the automatic method are lower than those of the other three, one can conclude that the automatic method is the most precise and has better repeatability and reproducibility.

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[2] Data from Edwards Air Force Base (F) appear to be completely different from the other laboratories and should not be part of the group. The variance and the standard deviation of the data from Livermore (G) are too high and should not be considered with the automatic ferrous sulfate method, but may be considered with the potentiometer ferrous sulfate method.

CONCLUSIONS

The automatic ferrous sulfate titration method can be considered equivalent to the nitrometer, and the repeatibility and reproducibility achieved by Hercules and RAAP (B and C) for this method are superior. RAAP can also achieve a repeatibility as good as or better than the nitrometer method using the potentiometer ferrous sulfate method.

The titanous chloride method has poor repeatibility but good reproducibility.

RECOMMENDATIONS

It is recommended that:

1. The automatic ferrous sulfate titration method be added to the JANNAF Propellant Characterization Working Group Handbook (ref 5) and to Specification Mil-N-244 as an alternate method for determining the percentage of nitrogen in nitrocellulose (ref 1).

2. Another round robin be conducted with the potentiometer ferrous sulfate titration method.

REFERENCES

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5. JANNAF Propellant Characterization Working Group Handbook (1 CRPG, 800.0 through 842), Chemical Propulsion Information Agency, Johns Hopkins University Applied Physics Laboratory, Laurel, MD, published annually.

6. Eric Bixon, "Statistical Guide for Determining the Equivalency of Two Chemical Test Methods," ARRADCOM, Dover, NJ, April 1982.

Table 1. Percentage of nitrogen in nitrocellulose lot 15340

Activities	Nitrometer			Titanous chloride			Automatic ferrous sulfate			Potentiometer ferrous sulfate				
	A	C	E	C	D	E	B	C	G	A	C	D	E	F
	13.12	13.16	13.11	13.05	13.17	12.99	13.15	13.15	13.18	13.23	13.13	13.16	13.19	12.25
	13.08	13.14	13.11	12.98	13.43	13.09	13.17	13.17	13.23	13.21	13.16	13.12	13.11	12.26
	13.09	13.14	13.12	13.00	13.22	13.00	13.14	13.18	13.17	13.23	13.17	13.17	13.18	12.21
	13.08	13.17	13.13	13.16	13.22	13.01	13.17	13.17	13.16	13.12	13.13	13.11	13.11	12.39
	13.02	13.16	13.12	13.02	13.22	13.13	13.18	13.16	13.12	13.17	13.15	13.16	13.10	12.68
	13.11	13.15	13.13	12.91	13.13	12.87	13.14	13.17	13.24	13.21	13.15	13.17	13.17	12.62
	13.09	13.14	13.10	13.30	13.19	13.19	13.13	13.16	13.15	13.21	13.15	13.18	13.17	12.72
	13.03	13.15	13.13	13.12	13.24	14.62	13.16	13.15	13.14	13.24	13.14	13.17	13.12	12.34
	13.08	13.17		13.34	13.14	12.94	13.16	13.16	13.08	13.22	13.13	13.18	13.09	12.44
	13.11	13.17		13.24	13.26	12.95	13.14	13.16	13.11	13.20	13.15	13.22	13.16	12.47
Mean	13.08	13.16	13.12	13.12	13.23	13.02	13.15	13.16	13.16	13.20	13.15	13.16	13.14	12.44
Variance	0.0009	0.0001	0.0001	0.0196	0.0064	0.0100	0.0004	0.0001	0.0025	0.0016	0.0001	0.0016	0.0016	0.0324
S.D.	0.03	0.01	0.01	0.14	0.08	0.10	0.02	0.01	0.05	0.04	0.01	0.04	0.04	0.18

Table 2. Percentage of nitrogen in nitrocellulose lot 30057

Activities	Nitrometer			Titanous chloride			Automatic ferrous sulfate			Potentiometer ferrous sulfate				
	A	C	E	C	D	E	B	C	G	A	C	D	E	F
	12.56	12.62	12.60	12.67	12.61	12.34	12.59	12.63	12.55	12.67	12.66	12.61	12.67	10.27
	12.55	12.61	12.59	12.67	12.65	12.10	12.63	12.62	12.59	12.70	12.66	12.61	12.60	10.14
	12.52	12.61	12.60	12.75	12.69	12.11	12.62	12.62	12.54	12.68	12.65	12.68	12.67	10.13
	12.53	12.62	12.61	12.77	12.66	12.11	12.60	12.62	12.66	12.69	12.61	12.65	12.69	10.45
	12.55	12.60	12.62	12.76	12.62	12.12	12.60	12.61	12.59	12.69	12.61	12.60	12.59	10.24
	12.56	12.60	12.60	12.76	12.62	12.38	12.59	12.64	12.63	12.67	12.65	12.68	12.69	10.06
	12.57	12.63	12.61	12.65	12.63	12.23	12.59	12.62	12.57	12.68	12.62	12.76	12.61	11.12
	12.56	12.63	12.64	12.75	12.62	11.90	12.62	12.62	12.57	12.64	12.64	12.88	12.66	10.71
	12.55	12.63		12.41	12.68	12.44	12.62	12.64	12.58	12.68	12.63	12.71		10.69
	12.56	12.60		12.71	12.68	12.40	12.63	12.62	12.58	12.66	12.65	12.69		10.78
Mean	12 55	12.62	12.61	12.69	12.65	12.21	12.61	12.62	12.59	12.68	12.64	12.69	12.65	10.46
Variance (ref 5)	0.0004	0.0001	0.0004	0.0121	0.0009	0.0289	0.0004	0.0001	0.0016	0.0004	0.0004	0.0064	0.0004	0.1225
S.D.	0.02	0.01	0.02	0.11	0.03	0.17	0.02	0.01	0.04	0.02	0.02	0.08	0.02	0.35

Table 3. Two-way analysis of variance (automatic and potentiometer) (ref 4)

Variance	Automatic titration[a]				Potentiometer titration[b]			
	SS[c]	DF[d]	MS[e]	F-test[f]	SS[c]	DF[d]	MS[e]	F-test[f]
Lots	2.9376	1	2.9376	32640.4445	3.7751	1	3.7851	905.1610
Activities	0.0014	1	0.0014	16.0000	0.0236	2	0.0118	2.8222
Interactions	0.0001	1	0.0001	0.4924	0.0084	2	0.0042	2.3702
Within	0.0066	36	0.0002	-	0.0952	54	0.0018	-
Totals	2.9457	39	-	-	3.9023	59	-	-
Repeatability (ref 6)			0.0118				0.0368	
Reproducibility (ref 6)			0.0242				0.0783	
Grand mean lot 15340			13.1585				13.1693	
Grand mean lot 30057			12.6165				12.6670	

[a] Hercules and RAAP (B and C)

[b] Olin Corporation, RAAP, and ARRADCOM (A, C; D)

[c] Sum of squares

[d] Degrees of freedom

[e] Mean square

[f] Variance of sample distribution

Table 4. Two-way analysis of variance (nitrometer and titanous chloride) (ref 4)

Variance	Nitrometer[a]				Titanous chloride[b]			
	SS[c]	DF[d]	MS[e]	F-test[f]	SS[c]	DF[d]	MS[e]	F-test[f]
Lots	2.7092	1	2.7092	712.4792	6.4419	1	6.4419	16.4190
Activities	0.0287	1	0.0287	7.8113	0.6687	2	0.3343	0.8522
Interactions	0.0038	1	0.0038	7.2085	0.7847	2	0.3923	7.0105
Within	0.0190	36	0.0005	-	3.0221	54	0.0559	-
Totals	2.7607	39	-	-	10.9174	59	-	-
Repeatability (ref 6)			0.0201				0.2702	
Reproducibility (ref 6)			0.1134				0.5280	
Grand mean lot 15340			13.1180				13.1717	
Grand mean lot 30057			12.5975				12.5163	

[a] Olin Corporation and RAAP (A and C)

[b] Hercules, RAAP, and Naval Ordnance Station (B, C; D)

[c] Sum of squares

[d] Degrees of freedom

[e] Mean square

[f] Variance of sample distribution

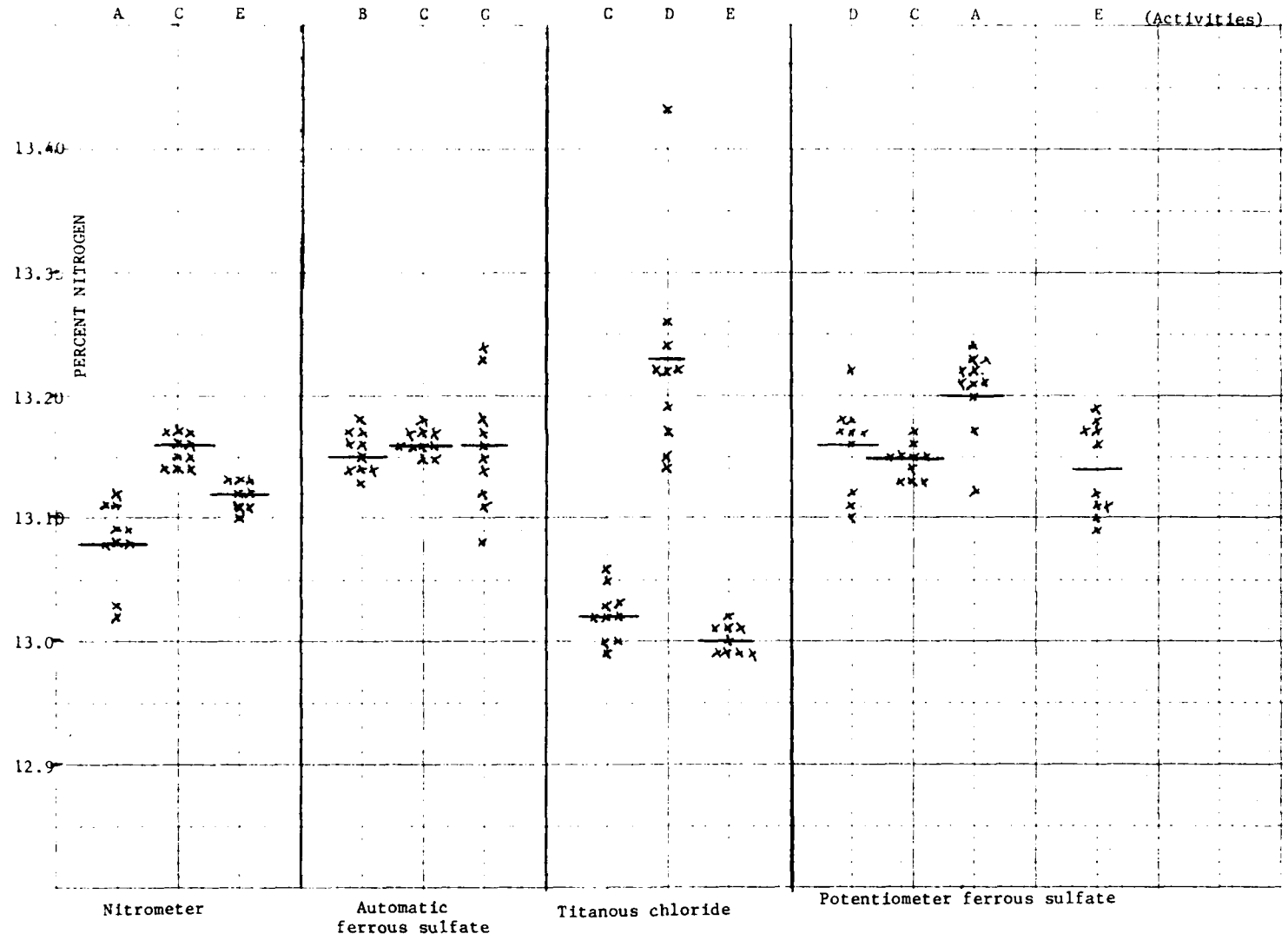

Figure 1. Percentage of nitrogen in nitrocellulose lot 15340

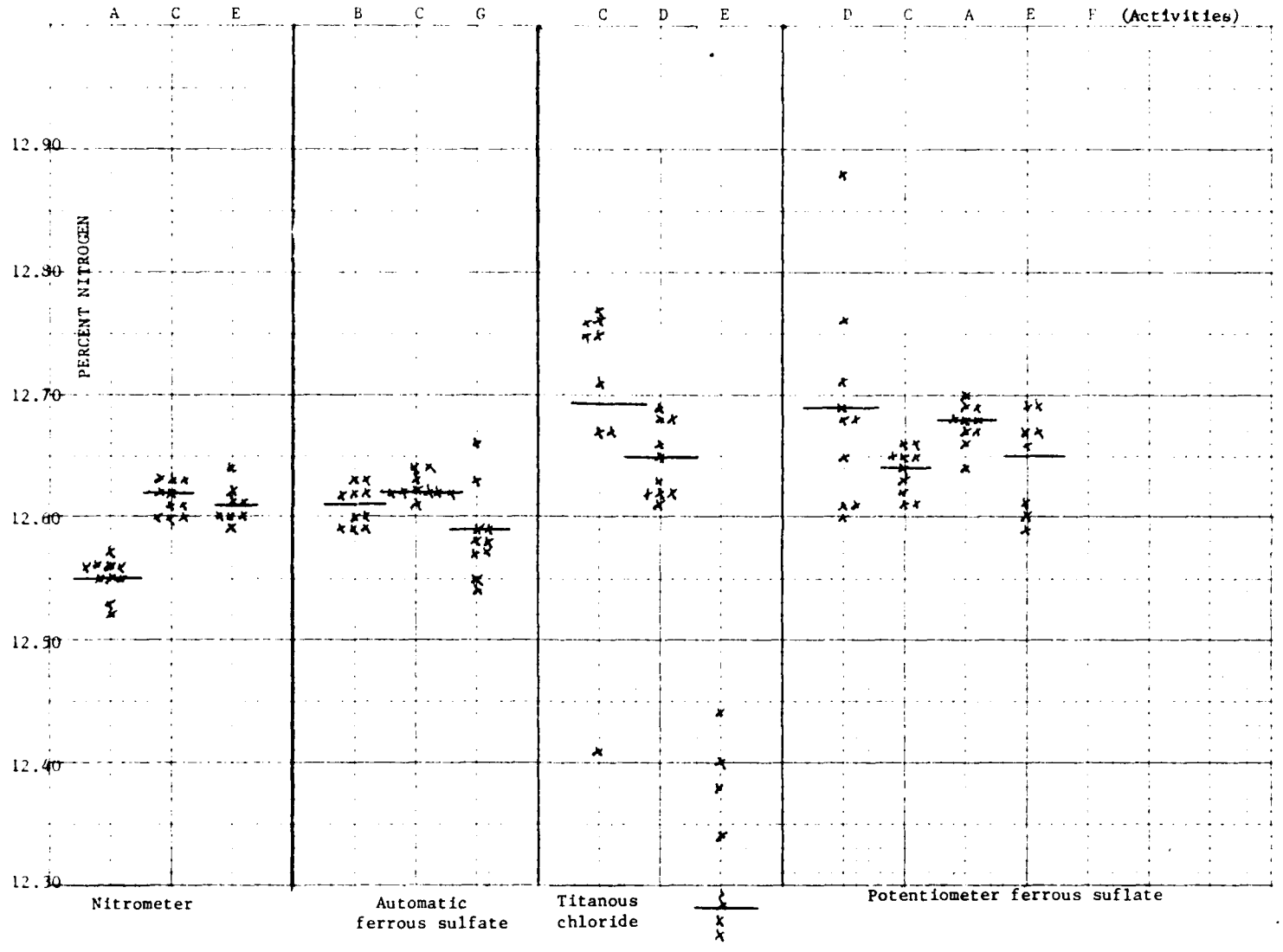

Figure 2. Percentage of nitrogen in nitrocellulose lot 30057

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